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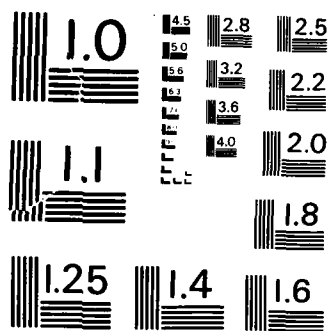
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DEVELOPMENT OF A TEST METHOD TO DETERMINE POTENTIAL PEROXIDE CONTENT IN TURBINE FUELS

INTERIM REPORT
BFLRF No. 199

By

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19. ABSTRACT (Continue on reverse if necessary and identify by block number) To develop a practical test method for the prediction of peroxide potential of fuels, experimental conditions were sought so that the oxidative tendencies of fuels could be assessed within a reasonable time, i.e., less than 48 hours. Through the generally accepted 43°C (110°F) bottle storage method of accelerated fuel aging, the relative ratings of four selected fuels' oxidative tendencies were established. A matrix of experiments was designed to allow selection of those short-term reaction conditions that would give results comparable to the 43°C bottle storage experiments. Additionally, the results of these experiments would allow the development of global reaction kinetics to aid the determination of fuel peroxidation potential. From a partially completed experimental matrix, fuel stressing was completed at 60° and 100°C under an initial oxygen pressure of 689 kPa (100 psig). Experimental results at 60°C gave inconclusive results. Results of the 100°C experiments produced essentially self-consistent results					
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19. ABSTRACT:

that also agreed with those of the bottle storage for the most stable and least stable fuels. Results of the two intermediate stability fuels, however, were interchanged.

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FOREWORD/ACKNOWLEDGEMENTS

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INTRODUCTION

Background

In 1976 the U.S. Navy experienced difficulties with A-7E aircraft that operated in the western Pacific. It was shown^{(1)*} that these difficulties were caused by peroxides in the turbine fuel that attacked the neoprene diaphragm in the engine's fuel pump. To avoid similar problems in the future, it would be advantageous to have a test method that would predict the maximum, or potential, peroxide content of a fuel even before the fuel has any measurable peroxide content. Thus far, attempts to predict fuel peroxidation tendencies were either too time consuming or produced false trends relative to ambient temperature results.

Objective

The objective of this study was to develop a practical test method for the prediction of the peroxide potential of fuels.

Approach

A study was made of the effects of oxygen concentration (pressure), temperature, and test duration on selected kerosene-type fuels. As indicators of the extent of fuel oxidation, oxygen uptake, peroxide content, formation of gum, water, and acidity were measured. These data were then compared to the results of a generally accepted long-term "bottle storage test" conducted at 43°C.

* Superscript numbers in parentheses indicate references listed at the end of this report.

II. TEST FUELS

Four kerosenes were selected to serve as "model" fuels for the initial phases of the program. One of these fuels (No. 0464) was a straight-run, salt-dried, and clay-treated, additive-free Jet A, to serve as a pristine, stable fuel. Two hydrocracked kerosenes were selected as potentially unstable fuels. Each of these unstable fuels was also percolated through a column of activated alumina to remove the peroxides and other polar components. Since one of the hydrocracked kerosenes (No. 11310) contained high concentrations of peroxides (420 ppm), it was only used after the alumina treatment. The other hydrocracked fuel (No. 11381) was used in the oxidation experiments both before and after the alumina treatment.

Chemical reactivity, including oxidizability of a fuel, depends upon the fuel's chemical composition. Thus, in addition to the ASTM-type analyses summarized in Table 1, a hydrocarbon-type analysis was also performed on each of the model fuels by ^1H and ^{13}C nuclear magnetic resonance (NMR) spectroscopy. Proton-type assignments were made according to the recommendations by Netzel and Hunter.⁽²⁾ Abbreviated NMR definitions are given in Table 2, while the spectroscopic data are summarized in Table 3.

It should be noted that the $\text{H}(\alpha-1)$ protons are reactive benzylic types, and that $\text{H}(\beta-\text{N})$ include naphthene hydrogens beta to an aromatic ring and hydrogens on tertiary carbon atoms. It was expected that fuels with widely different oxidative tendencies would exhibit gross differences in their NMR spectra, especially in their $\text{H}(\alpha-1)$ and $\text{H}(\beta-\text{N})$ regions. Thus far, this expectation has not been fulfilled. The concentrations of reactive sites in Table 3 as measured by NMR do not reflect the relatively large differences in the reactivity of the test fuels with oxygen.

TABLE 1. ANALYSIS OF SELECTED KEROSENES

Property	ASTM Designation	Fuel Number			JP-5 Spec (partial) MIL-T-5624-L
		0464(*)	11310(**)	11381(**)	
Specific Gravity @ 15.5°C	D 1298	0.8185	0.7976	0.8174	0.788-0.845
Flash Point, °C	D 93	45	47	66	60 min
Freeze Point, °C	D 2386	-41	-44	-40	-46 max
Smoke Point, mm	D 1322	19.2	24.7	20.8	19.0 min
Sulfur, wt%	D 2622	0.03	0.01	0.01	
Hydrocarbon Types, vol%	D 1319				
Saturates		80.7	87.8	78.8	
Olefins		1.4	0.5	0.7	5.0 max
Aromatics		17.9	11.7	20.5	25.0 max
Distillation	D 86				
Initial Boiling Point, °C		150	150	181	
10% Recovered, °C		182	166	198	205 max
50% Recovered, °C		219	192	219	
90% Recovered, °C		256	244	253	
End Point, °C		284	281	273	290 max
Heat of Combustion, Gross, Btu/lb	D 240	19,691	20,134	19,700	
Heat of Combustion, Net, Btu/lb	D 240	18,437	18,851	18,442	18,300 min
Oxidation Stability, 16 hr	D 873				
Soluble gum, mg/100 mL		3.4	90.5	6.8	
Insoluble gum, mg/100 mL		0.0	0.9	0.4	
Precipitate, mg/100 mL		0.0	0.7	0.7	
Total potential residue		3.4	92.1	7.9	
Peroxide Number, ppm	D 3703	0.0	420	2.8	

* Straight-run, salt-dried, and clay-treated additive-free Jet A

** Hydrocracked kerosene

TABLE 2. PROTON NMR DEFINITIONS

<u>Symbol</u>	<u>Definitions</u>	<u>Chemical Shift Range (ppm from TMS)</u>
H(AR)	= Total Aromatic Hydrogens	6.6-8.3
H(OL)	= Olefinic Hydrogens	4.0-6.0
H(α -1)	= Methylene Hydrogens Alpha to Aromatic Ring	2.3-4.0
H(α -2)	= Methyl Hydrogens Alpha to Aromatic Ring	1.9-2.3
H(β -N)	= Naphthene Hydrogens Beta to Aromatic Ring and Hydrogen on Tertiary Carbon Atoms	1.6-1.9
H(β)	= β -CH ₂ - and β -CH ₃ to Aromatic Ring and Normal Alkane -CH ₂ -Hydrogens	1.0-1.6
H(γ)	= γ -CH ₃ to Aromatic Ring and Normal or Branch Alkane -CH ₃ Hydrogens	0.5-1.0

Source: Reference 2.

TABLE 3. HYDROGEN-TYPE ANALYSIS BY PROTON NMR SPECTROSCOPY

<u>H Type</u>	<u>Percent Hydrogen Type in Fuels</u>			
	<u>11310/Al₂O₃</u>	<u>11381</u>	<u>11381/Al₂O₃</u>	<u>0464</u>
H(AR)	2.4	3.0	2.9	2.7
H(OL)	0.0	0.0	0.0	0.0
H(α -1)	1.0	3.2	3.4	3.2
H(α -2)	1.9	4.3	4.3	4.8
H(β -N)	4.8	6.2	5.3	4.8
H(β)	50.1	50.5	50.2	48.9
H(γ)	39.8	32.8	33.8	35.5

III. APPARATUS AND PROCEDURES

To establish baseline data on the long-term stability of the four selected "model" fuels, the generally accepted method of bottle storage at 43°C⁽³⁾ was used. In this procedure, 200 mL of each of the test fuels were saturated with "synthetic" air (21% O₂ and 79% N₂, purified) at 300 mL per minute for 5 minutes. Then the fuel samples were stored for an extended time in the dark at 43°C in sealed 500-mL amber borosilicate bottles. After aging periods of 1, 2, 3, and 4 weeks, followed by 4-week intervals, one bottle of each fuel was retrieved for analysis. The oxygen content was determined by GC in both the liquid and vapor phases, in addition to measurement of peroxides, gum, water, and acid number of the liquid phase. If the oxygen concentration in the vapor phase was below 10.0 vol%, the remaining bottles of the same fuel were again aerated, as described above.

Experimental accelerated oxidative stressing of these fuels was done in a pressure reactor. The reactor was made of Type 316 stainless steel equipped with a magnetically-coupled stirrer driven by a variable speed motor, an external electrical heating element, and an internal cooling loop with automatic temperature control. Valves and fittings permit introduction of gases and liquids and withdrawal of samples either at the bottom or the top of the vessel. The sample is held in a borosilicate reactor liner. Additionally, the reactor is equipped with a 1380 kPa (200 psig) rupture disc, and a 1380 kPa (200 psig) recording pressure gauge, both of which are replaceable with items rated at pressures up to 13,790 kPa (2000 psig). In laboratory practice, the borosilicate reactor liner is charged with 300 mL of fuel. The assembled system is then purged with oxygen of ultra-high purity (99.99% min), followed by pressurization to a predetermined pressure, e.g., 689 kPa (100 psig). While the fuel is stirred at about 150 rpm, the fuel temperature is raised to the test temperature within 20-30 minutes, and held there for the test duration within a tolerance of $\pm 0.5^\circ\text{C}$. The pressure and temperature within the reactor are continuously recorded to allow the calculation of the amount of oxygen that reacted with the fuel. Upon completion of the experiment, the reactor is disassembled at room temperature and atmospheric pressure. All the interior parts are washed, in several increments, with a total of 30 mL of 2-propanol to collect and dissolve all the formed water. The formed gum is dissolved in minimum measured amounts of an equivolume mixture of toluene, acetone, and methanol (TAM). These washings are added to the fuel that is analyzed for peroxides (ASTM D 3703), gum (ASTM D 381), water (ASTM D 1744), and acid number (ASTM D 3242).

IV. RESULTS AND DISCUSSION

Bottle storage of fuels under atmospheric air at 43°C established the ranking of the four fuels in order of their expected storage stability. The experimental data are summarized in Table 4. The same data are graphically presented in Figures 1 through 5, in which time dependence is illustrated for oxygen consumption in the vapor and liquid phases, as well as that of the generation of peroxides, water, and gum, respectively. Within experimental error, each of the variables give the same ranking of fuels' storage stability, that is, the order of decreasing stability is: Fuel Nos. 0464, 11381, alumina-treated 11310, and alumina-treated 11381.

It is a generally accepted approximation that storage for about 12 weeks at 43°C has about the same deteriorating effect on a fuel as does a full year of storage under ambient temperature conditions. Since such a lengthy procedure is not suitable for quality control, the goal of this project was to find accelerated fuel-aging conditions that yield comparable results. It was a further goal to design the experimental matrix in such a way that the results would be usable for a chemical kinetics treatment. In the experimental matrix, a cross-correlation was sought among time, temperature, and oxygen pressure effect as summarized:

<u>Temperature, °C</u>	<u>Pressure, psig</u>		
60	50	100	150
80	50	100	150
100	50	100	150

In this report, experimental results of fuel oxidations are described under the influence of 689 kPa (100 psig) oxygen at 60° and 100°C for stress durations up to 48 hours, as summarized in Tables 5 and 6, respectively. Corresponding graphical illustrations of data are given in Figures 6 through 9 and 10 through 13, respectively.

Data at 60°C were obtained only after 24- and 48-hour stress periods. Oxygen depletion during the 60°C experiments, as measured by the pressure gauge, was insignificant. Oxygen uptake, as calculated from pressure gauge readings at room temperature before and after the experiments, leveled off at a low value of about 19.2 mmole/L of fuel (with a standard deviation of 1.8). The peroxide buildup paralleled the results obtained during the bottle storage. However, the quantities of

TABLE 4. BOTTLE STORAGE OF KEROSENES UNDER AIR AT 43°C

Fuel No.	Storage, weeks	O ₂ in Vapor Phase, vol %	Liquid Phase			H ₂ O, ppm
			O ₂ , ppm	Peroxides, ppm	S.J. Gum, mg/100 mL	
464	0	20.2	68	0	0.2	--
	1	20.7	61	0	3.8	--
	2	20.2	62	0	0.0	--
	3	20.6	61	0	1.9	--
	4	20.4	58	0	0.5	--
	8	20.7	64	0	0.3	--
	12	20.3	57	2	1.3	59
	16	20.4	45	2	1.2	41
	20	20.2	59	2	0.7	36
	24	20.0	61	2	0.9	51
11310/Al ₂ O ₃	0	20.6	70	0	0.6	--
	1	21.0	55	5	3.4	--
	2	20.0	71	14	0.2	--
	3	19.9	62	26	0.8	--
	4	18.6	57	54	0.5	--
	8	16.2	53	124	0.5	--
	12	11.9	33	243	0.4	67
	16*	6.1	0	380	1.0	62
	20	13.6	43	599	0.6	74
11381 "As Is"	24	4.8	5	841	0.6	159
	0	20.6	72	1	2.6	--
	1	20.6	59	3	5.9	--
	2	20.5	60	2	1.6	--
	3	20.9	58	2	1.7	--
	4	20.6	59	4	0.9	--
	8	20.6	61	8	0.4	--
	12	20.4	60	5	1.2	43
	16	19.4	41	34	1.7	32
11381/Al ₂ O ₃	20	20.2	59	16	0.8	59
	24	7.6	5	362	2.0	59
	0	20.6	69	0	1.0	--
	1	20.7	54	2	4.4	--
	2	21.3	63	6	0.8	--
	3	20.3	59	11	1.1	--
	4	19.9	50	21	0.4	--
	8	14.6	42	137	0.3	--
	12*	4.2	0	386	1.4	72
11381/Al ₂ O ₃	16*	8.2	0	610	2.6	79
	20*	4.3	6	1119	2.7	167
	24	4.8	0	1471	6.1	258

*Reaerated

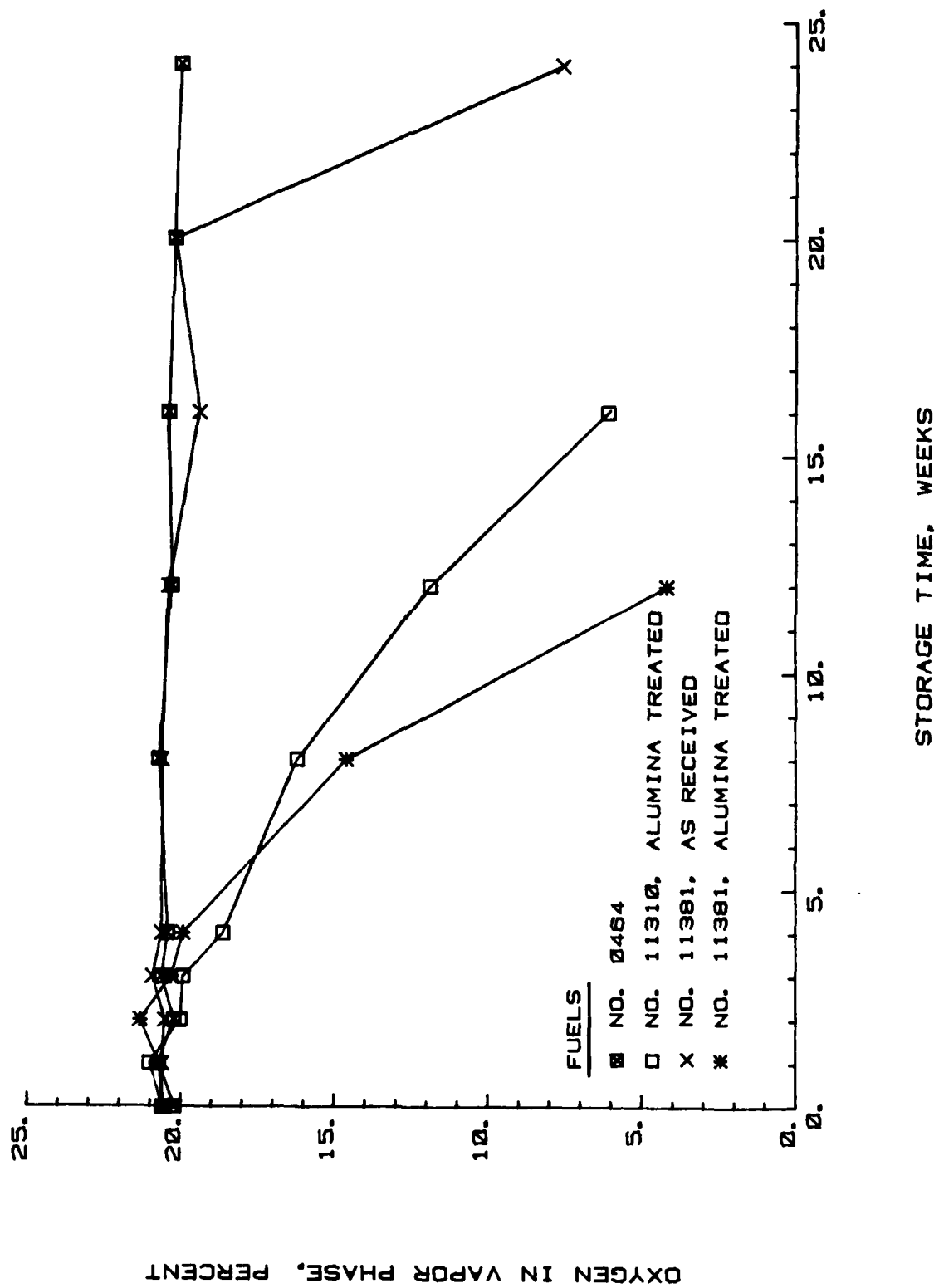


FIGURE 1. BOTTLE STORAGE UNDER AIR AT 43°C (OXYGEN IN VAPOR PHASE, PERCENT)

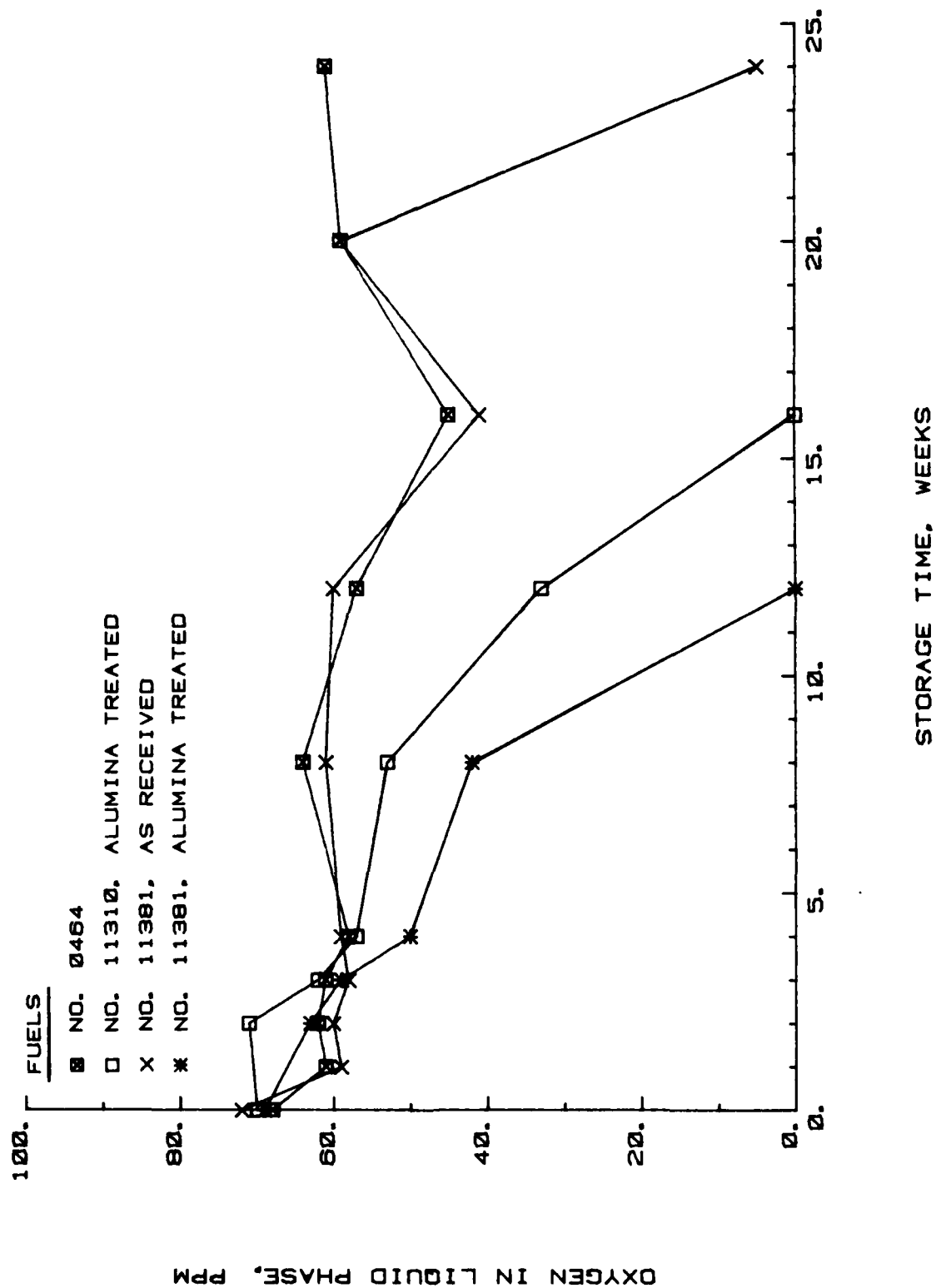


FIGURE 2. BOTTLE STORAGE UNDER AIR AT 43°C (OXYGEN IN LIQUID PHASE, PPM)

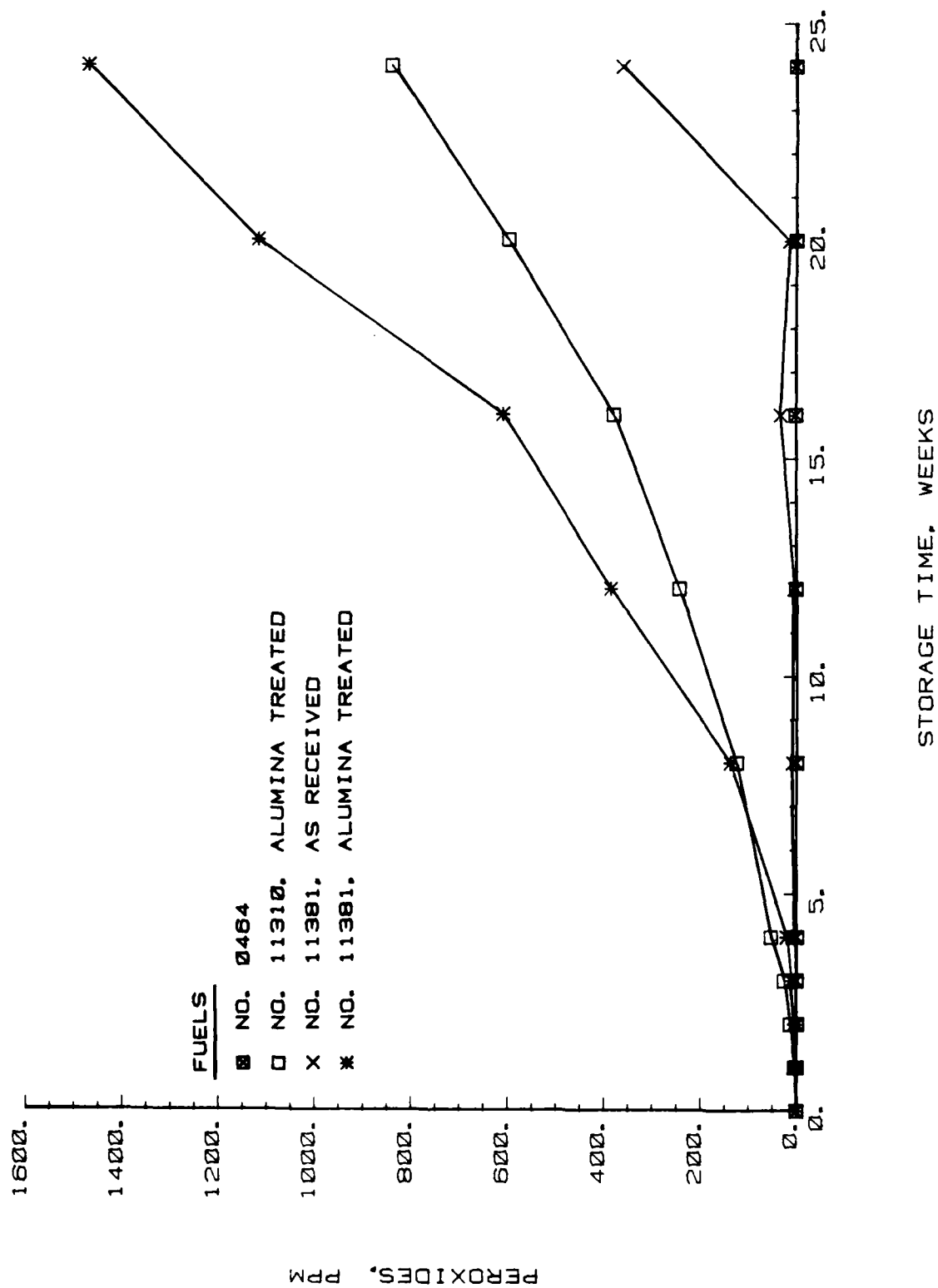


FIGURE 3. BOTTLE STORAGE UNDER AIR AT 43°C (PEROXIDES, PPM)

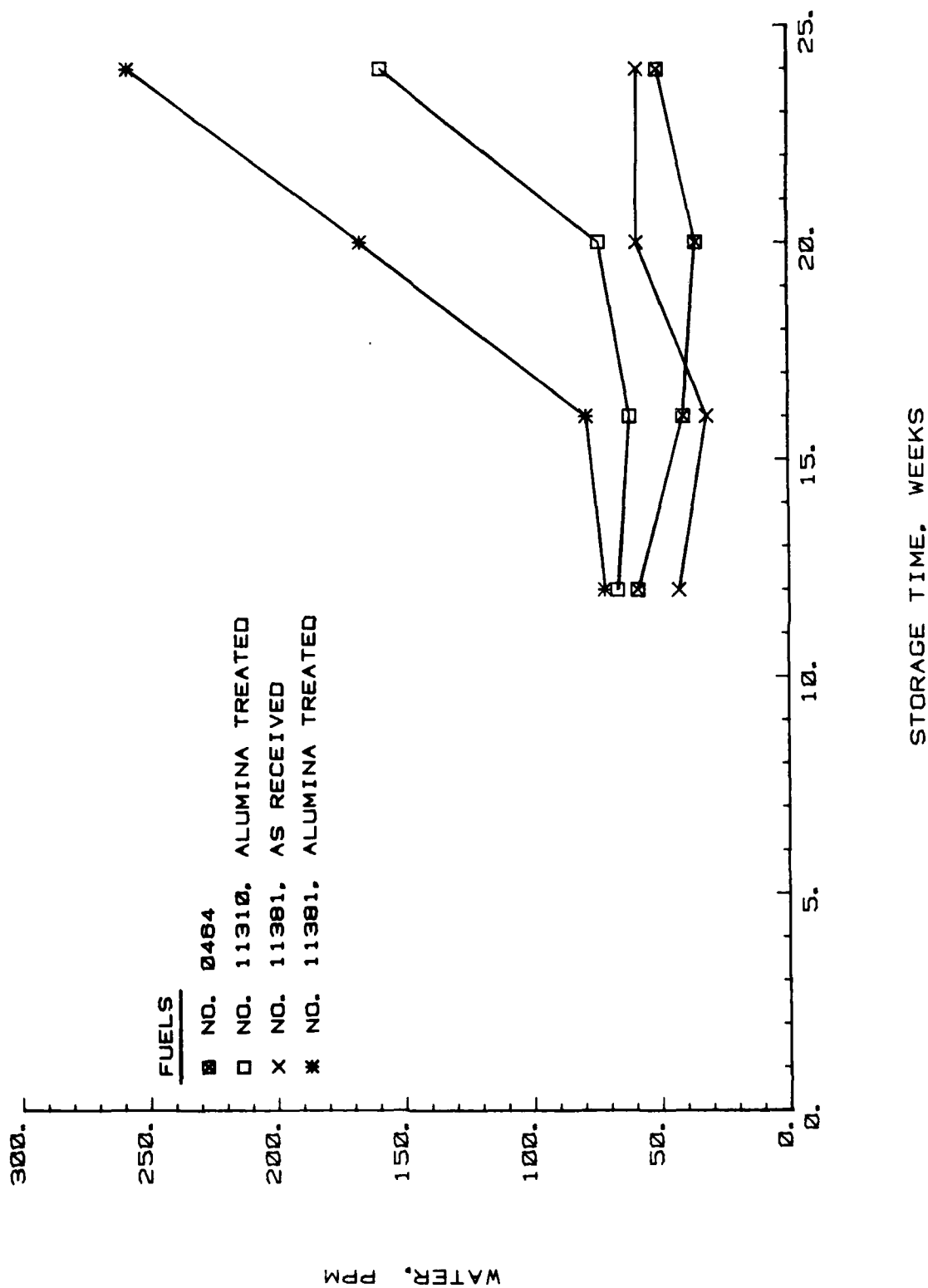


FIGURE 4. BOTTLE STORAGE UNDER AIR AT 43°C (WATER, PPM)

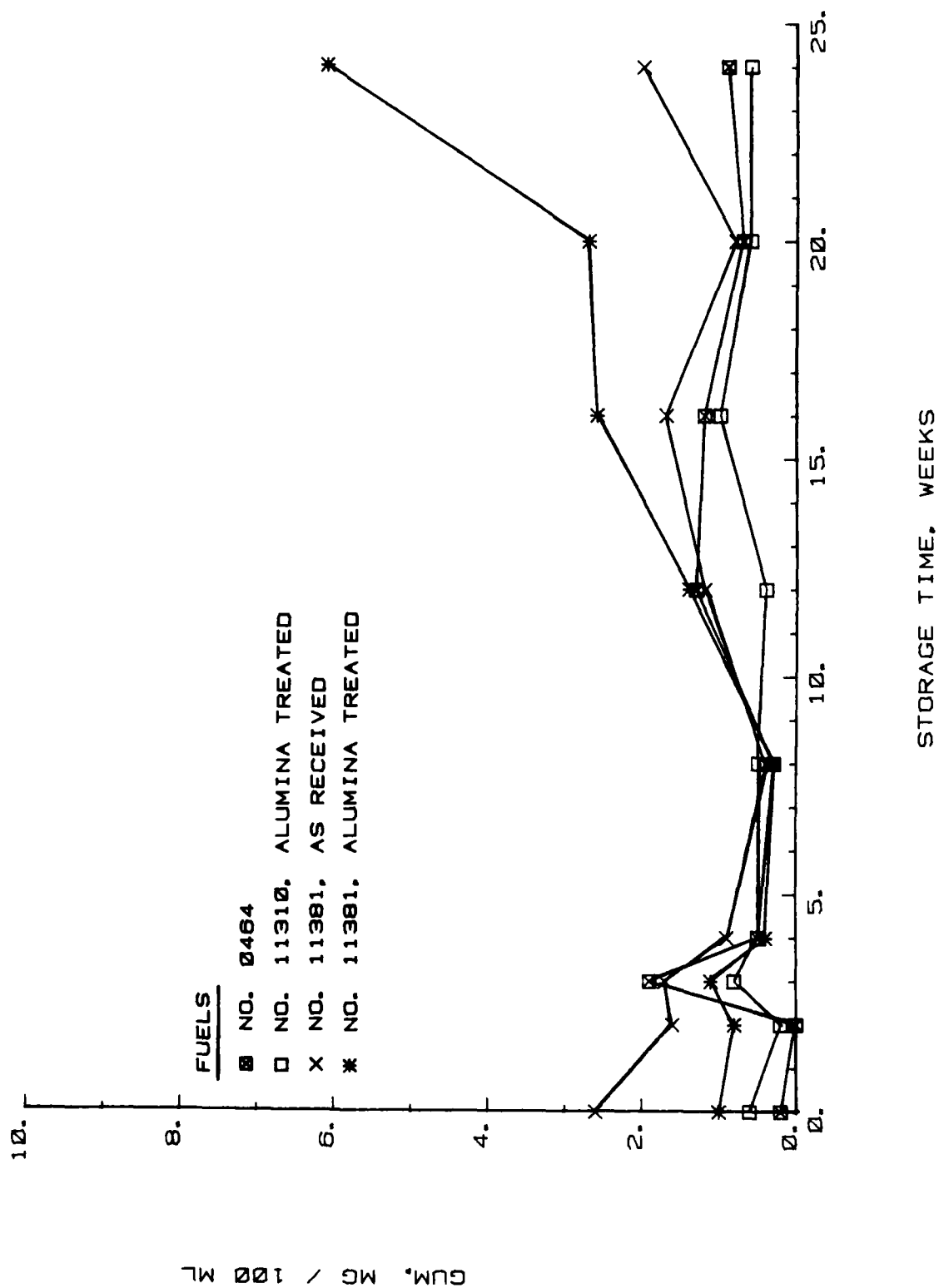


FIGURE 5. BOTTLE STORAGE UNDER AIR AT 43°C (GUM, MG/100 ML)

TABLE 5. OXIDATION OF KEROSENES AT 60°C UNDER AN INITIAL OXYGEN PRESSURE OF 100 PSIG

Fuel No.	Date	Time, hr	Oxygen Used, mmole/L	Peroxides ppm	S.I. Gum, mg/100mL	Water, ppm	TAN, mg KOH/g	Oxygen Depletion (psig) Vs Time (hours)													
								1	2	4	8	12	16	20	24	28	32	36	40	44	48
464	01-15-85	0	0	0.0	0.3	42	<0.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--
464	02-21-85	24	17.0	0.0	0.7	204	<0.1	100	100	100	100	100	100	100	100	100	100	100	100	100	100
464	03-11-85	48	20.9	2.0	0.6	220	<0.1	98	98	98	98	98	98	98	98	98	98	98	98	98	98
11310/Al.O.	01-15-85	0	0	0.0	0.0	26	<0.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--
11310/Al.O.	02-20-85	24	18.3	2.5	0.0	229	<0.1	100	100	100	100	100	100	100	100	100	100	100	100	100	100
11310/Al.O.	03-06-85	48	18.1	6.7	0.0	198	<0.1	100	100	100	100	100	100	100	100	100	100	100	100	100	100
11381	01-15-85	0	0	1.0	1.8	29	<0.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--
11381	02-14-85	24	21.1	3.8	1.7	115	<0.1	98	98	98	98	98	98	98	98	98	98	98	98	98	98
11381	02-04-85	48	20.8	2.0	4.5	150	<0.1	99	99	99	99	99	99	99	99	99	99	99	99	99	99
11381/Al.O.	01-15-85	0	0	0.0	0.8	9	<0.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--
11381/Al.O.	02-12-85	24	16.9	2.0	0.5	161	<0.1	101	101	101	101	101	101	101	101	101	101	101	101	101	101
11381/Al.O.	02-26-85	48	20.2	10.3	1.3	175	<0.1	99	99	99	99	99	99	99	99	99	99	99	99	99	99

Al.O. Fuel percolated through alumina

TABLE 8. OXIDATION OF KEROSENE AT 100°C UNDER AN INITIAL OXYGEN PRESSURE OF 100 PSIG

Fuel No.	Date	Time, hr	Oxygen Used, mmole/L	Peroxides, ppm	S.J.Gum, mg/100mL	Water, ppm	TAN, mg KOH/g	Oxygen Depletion (psi) Vs Time (hours)															
								1	2	3	4	6	8	10	12	14	16	18	20	22	24		
0464		0	0	0	0.2	42	<0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0464	10-31-84	6	17.6	2.8	0.8	56	—	108	108	108	108	108	—	—	—	—	—	—	—	—	—	—	—
0464	10-15-84	24	18.0	4.5	0.5	—	—	110	110	110	110	110	110	110	110	110	110	110	110	110	110	110	110
11310/Al.O.		0	0	0	0.6	29	<0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11310/Al.O.	11-12-84	4	25.4	44	0.5	28	<0.1	108	108	107	107	—	—	—	—	—	—	—	—	—	—	—	—
11310/Al.O.	10-23-84	6	23.0	60	0.5	40	—	110	110	110	110	109	—	—	—	—	—	—	—	—	—	—	—
11310/Al.O.	01-24-85	16	24.4	263	2.5	342	—	115	115	115	115	114	113	111	109	107	105	—	—	—	—	—	—
11310/Al.O.	10-17-84	24	99.3	949	2.7	450	—	111	111	111	111	109	107	103	98	93	88	83	77	71	65	—	—
11381		0	0	0	2.6	40	<0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381	01-11-85	6	9.3	49	8.5	112	—	116	116	116	116	116	—	—	—	—	—	—	—	—	—	—	—
11381	01-31-85	6	11.7	34	2.3	132	<0.1	112	112	112	112	112	—	—	—	—	—	—	—	—	—	—	—
11381	Average	6	10.5	42	5.4	122	<0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381	St. Dev.	0	1.7	11	4.4	14	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381	01-08-85	10	20.4	146	10.9	257	—	111	111	111	111	110	110	108	—	—	—	—	—	—	—	—	—
11381	01-10-85	10	19.9	96	7.4	197	0.2	113	113	113	113	112	111	108	—	—	—	—	—	—	—	—	—
11381	Average	10	20.2	121	9.2	227	0.2	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381	St. Dev.	0	0.4	35	2.5	42	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381	11-28-84	16	57.0	648	3.8	148	0.1	107	107	107	107	107	106	105	102	97	88	—	—	—	—	—	—
11381	01-28-85	16	52.8	851	26.6	421	—	112	112	112	112	112	111	109	105	98	89	—	—	—	—	—	—
11381	Average	16	54.9	750	15.2	285	0.2	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381	St. Dev.	0	3.0	144	16.1	193	0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381	10-02-84	24	137.0	1900	56.0	—	—	109	109	109	109	109	108	106	103	98	91	81	68	56	43	—	—
11381	10-05-84	24	128.6	2028	50.5	—	—	108	108	108	108	108	107	106	103	99	93	84	73	60	47	—	—
11381	11-26-84	24	151.7	1941	70.0	936	0.6	109	109	109	109	109	108	106	103	97	87	75	60	46	34	—	—
11381	01-15-85	24	153.8	2397	—	486	—	111	111	111	111	111	109	106	102	94	84	71	57	41	33	—	—
11381	Average	24	142.8	2067	58.8	711	0.6	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381	St. Dev.	0	12.1	227	10.1	318	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381	01-16-85	48	236.6	2080	598	3295	5.3	(110)	(105)	(102)	(99)	(83)	(73)	(65)	(56)	(49)	(43)	(37)	(32)	(27)	(23)	—	—
11381/Al.O.		0	0	0	1.0	37	<0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	11-07-84	4	26.7	91	1.1	32	<0.1	106	106	106	106	—	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	10-29-84	6	29.7	209	0.7	—	—	106	106	107	106	104	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	12-06-84	6	25.4	145	1.1	15	<0.1	108	108	107	107	106	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	12-17-84	6	26.7	120	0.5	23	<0.1	108	108	108	107	106	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	Average	6	27.3	158	0.8	19	<0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	St. Dev.	0	2.2	46	0.3	6	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	11-30-84	10	46.5	114	0.2	29	<0.1	108	108	108	108	108	108	107	—	—	—	—	—	—	—	—	—
11381/Al.O.	12-18-84	10*	35.7	387	2.2	39	<0.1	107	107	107	107	106	103	99	—	—	—	—	—	—	—	—	—
11381/Al.O.	Average	10	41.1	251	1.2	34	<0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	St. Dev.	0	7.6	193	1.4	7	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	12-19-84	16*	96.9	1400	13.5	509	0.2	107	107	107	106	104	101	96	89	79	68	—	—	—	—	—	—
11381/Al.O.	01-03-85	16*	115.8	1479	32.1	835	0.4	107	107	106	105	102	97	89	79	67	55	—	—	—	—	—	—
11381/Al.O.	01-30-85	16*	77.5	1381	15.7	631	—	112	112	112	112	112	111	109	105	98	89	—	—	—	—	—	—
11381/Al.O.	Average	16	96.7	1420	20.4	658	0.3	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	St. Dev.	0	19.2	52	10.2	165	0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	10-08-84	24	153.1	1613	76.3	—	—	112	112	112	111	110	106	102	96	87	77	66	54	43	33	—	—
11381/Al.O.	10-10-84	24	166.8	1749	70.7	—	—	111	110	110	110	109	106	100	93	83	72	60	48	36	24	—	—
11381/Al.O.	12-19-84	24*	150.6	1872	57.2	1264	<0.1	111	111	111	111	109	106	103	98	91	82	71	59	46	35	—	—
11381/Al.O.	Average	24	156.8	1745	68.1	1264	0.2	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
11381/Al.O.	St. Dev.	0	8.7	130	9.8	—	0.1	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—

Al.O. Fuel percolated thru alumina

* Second batch of fuel percolated through activated neutral alumina

() System repressurized after first 24 hours of oxidation. Pressure readings taken during the second 24 hours of stress.

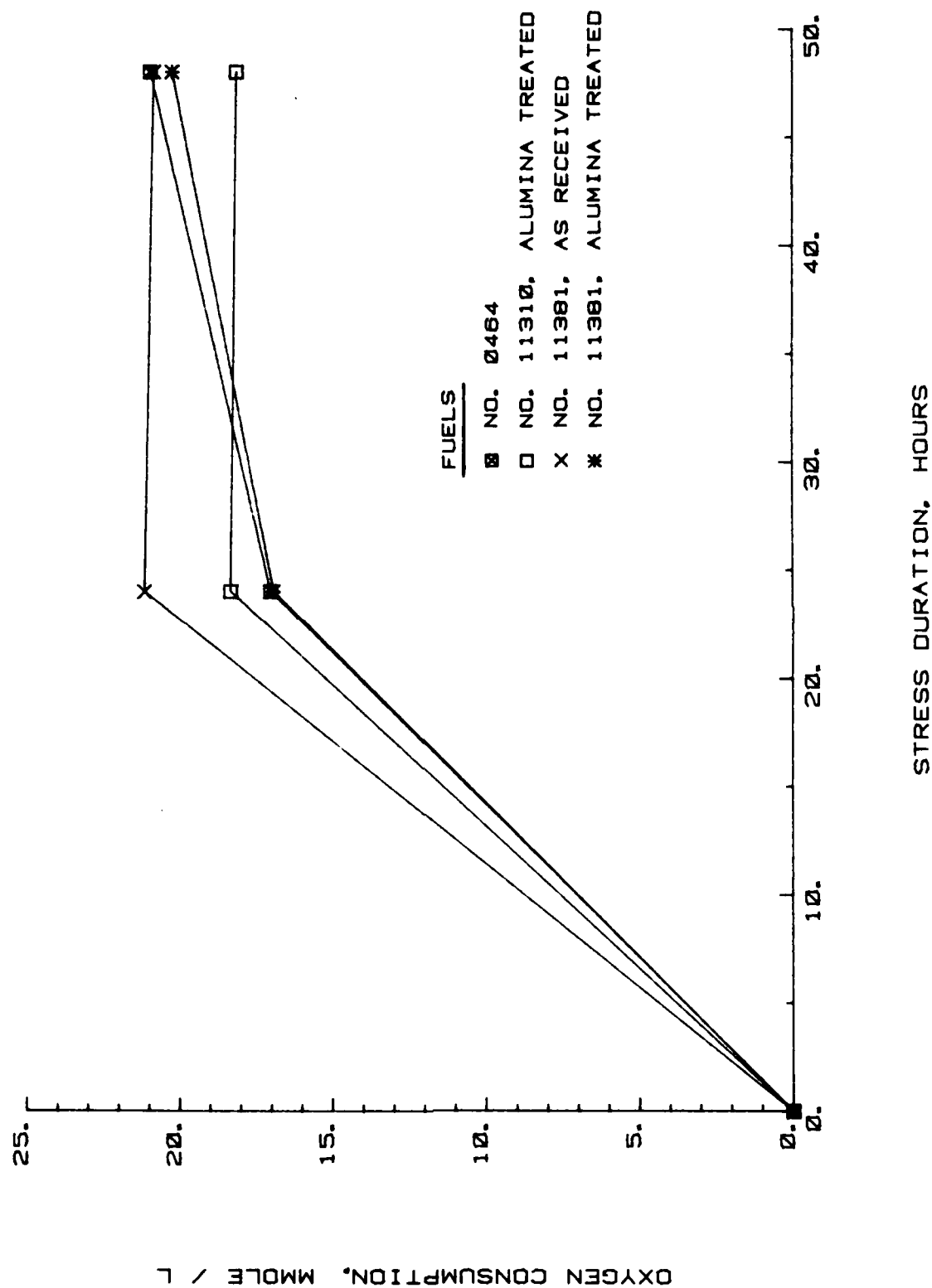


FIGURE 6. OXIDATION AT 60°C WITH 100 PSI OXYGEN (OXYGEN CONSUMPTION, MMOL/L)

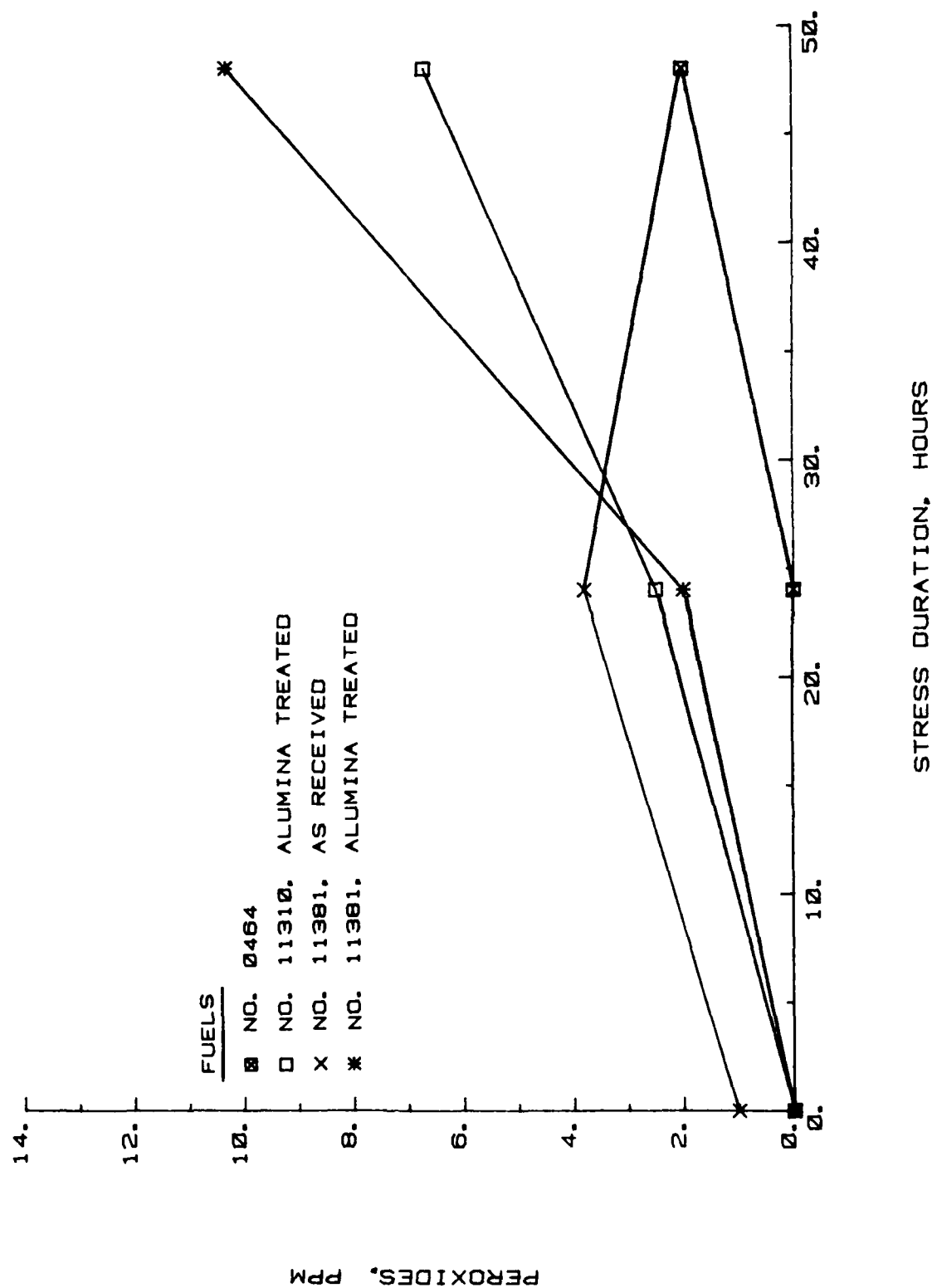


FIGURE 7. OXIDATION AT 60°C WITH 100 PSI OXYGEN (PEROXIDES, PPM)

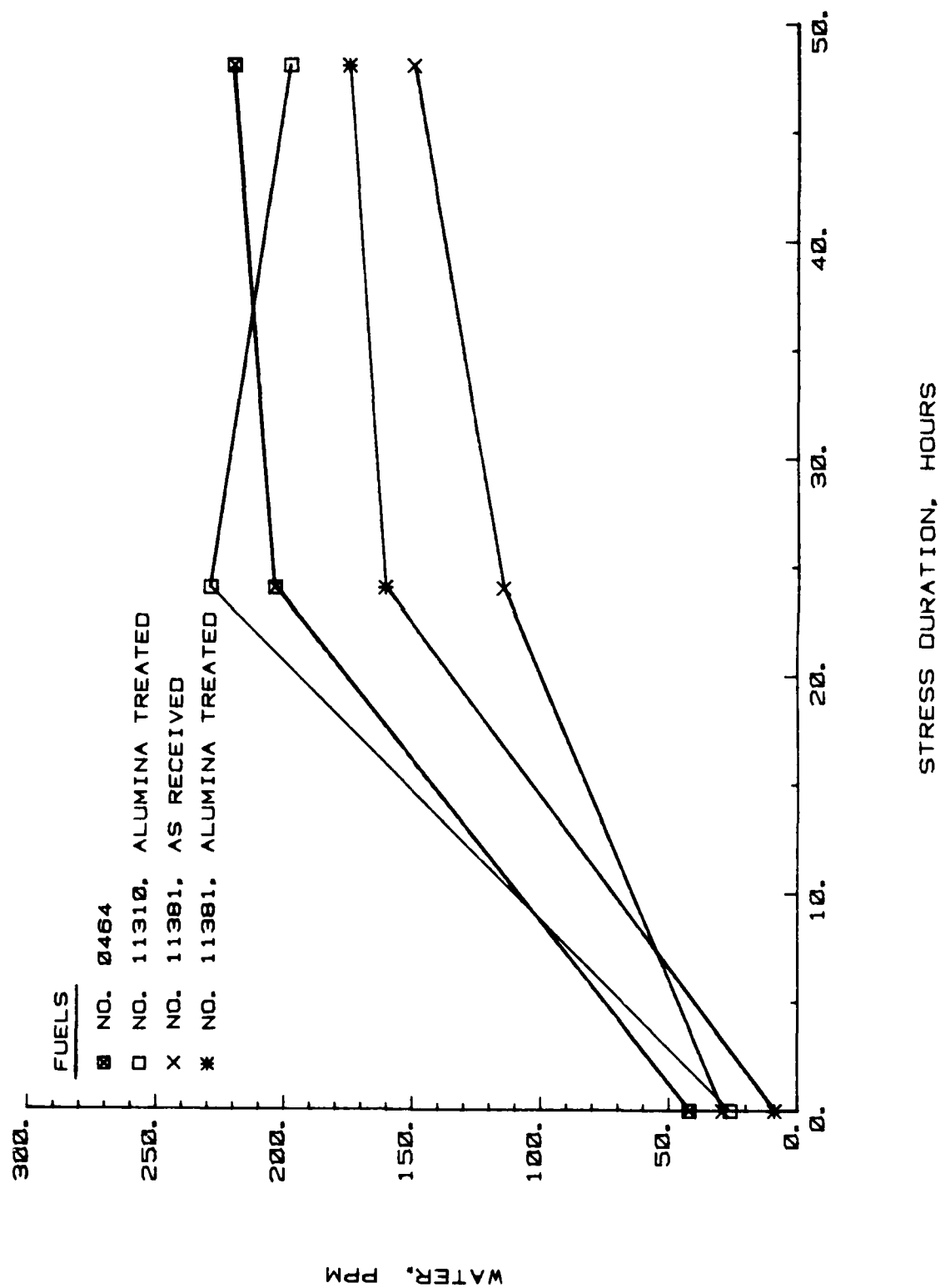


FIGURE 8. OXIDATION AT 60°C WITH 100 PSI OXYGEN (WATER, PPM)

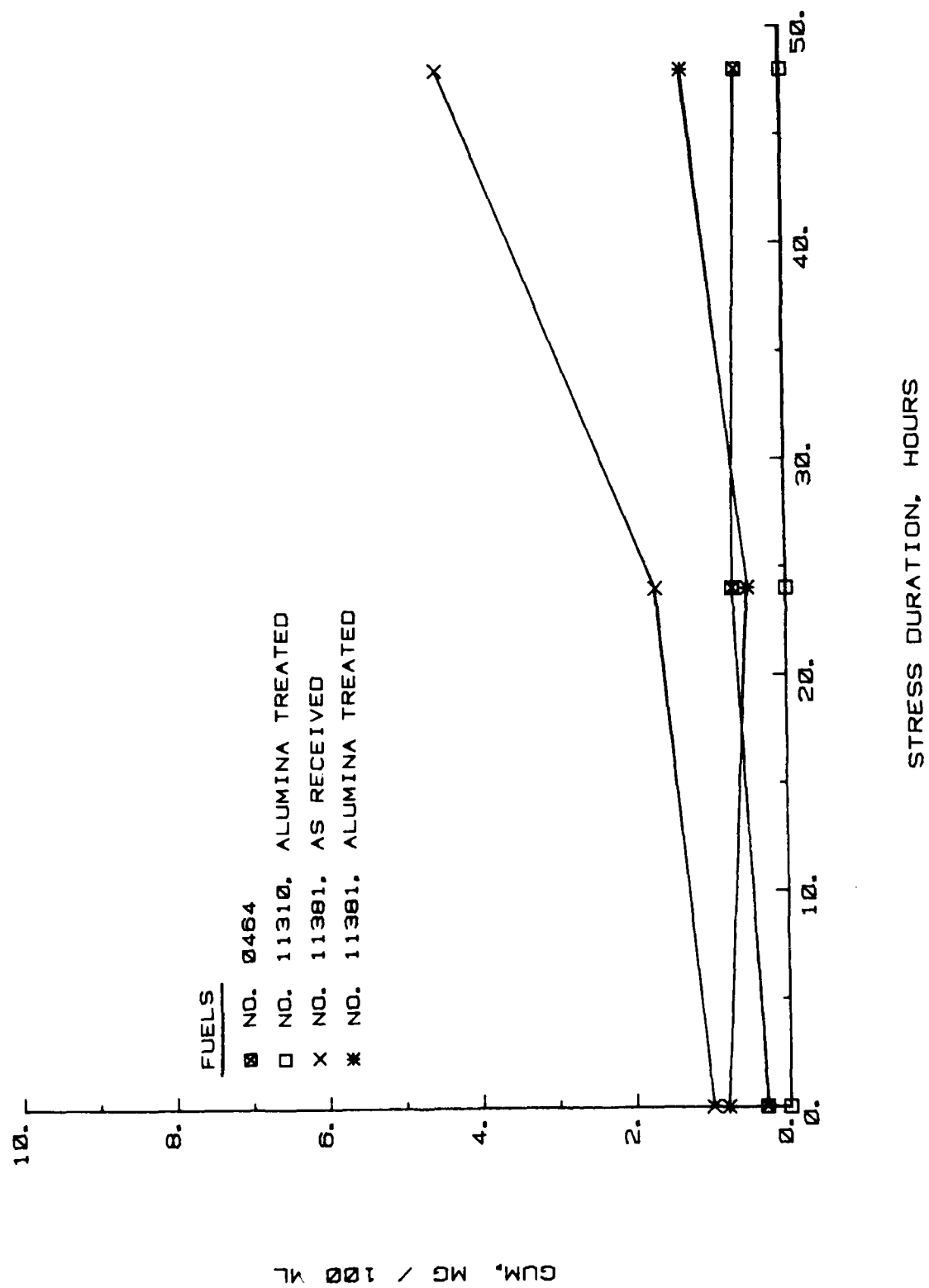


FIGURE 9. OXIDATION AT 60°C WITH 100 PSI OXYGEN (GUM, MG/100 ML)

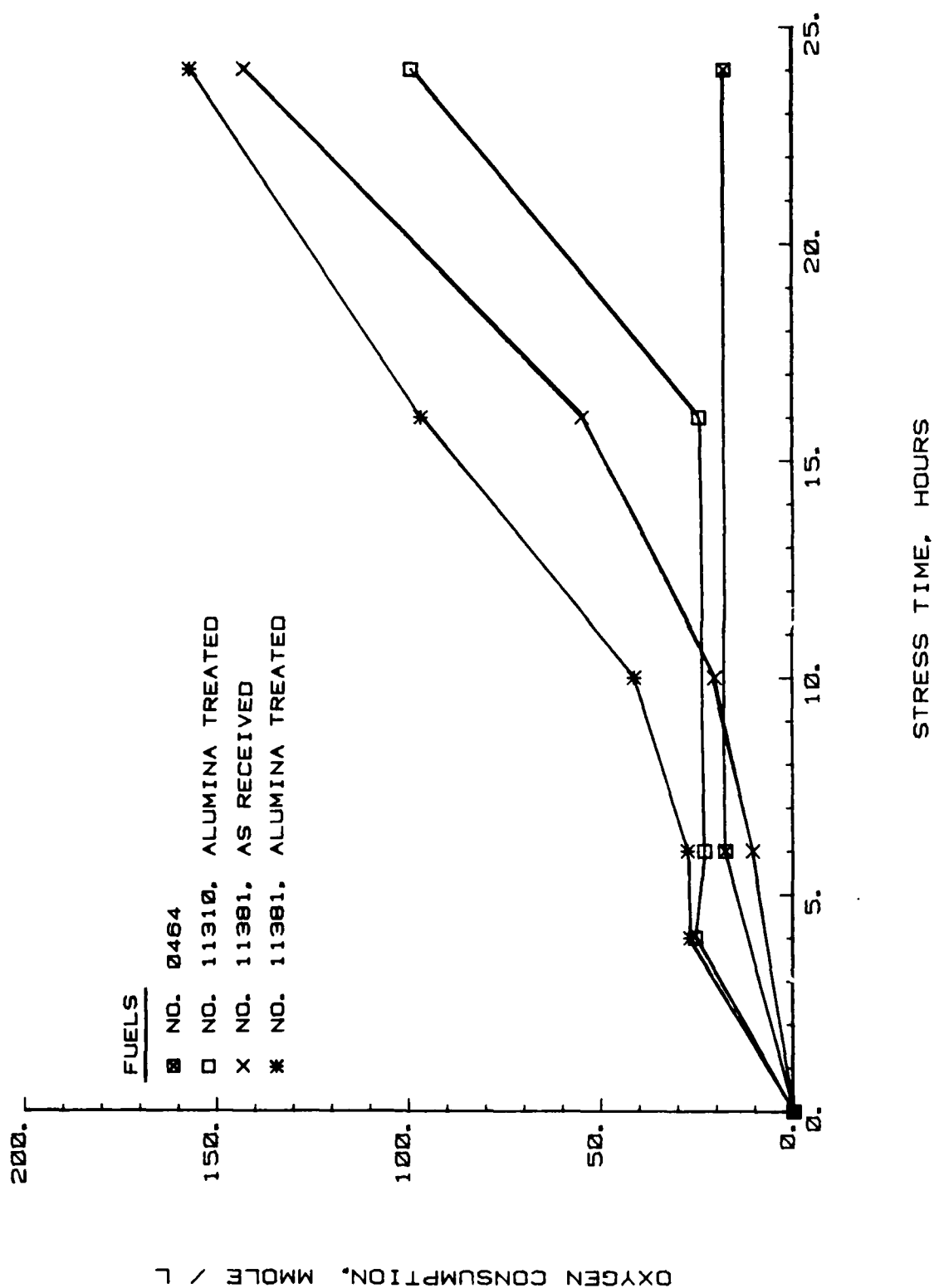


FIGURE 10. OXIDATION AT 100°C WITH 100 PSI OXYGEN (OXYGEN CONSUMPTION, MMOLE/L)

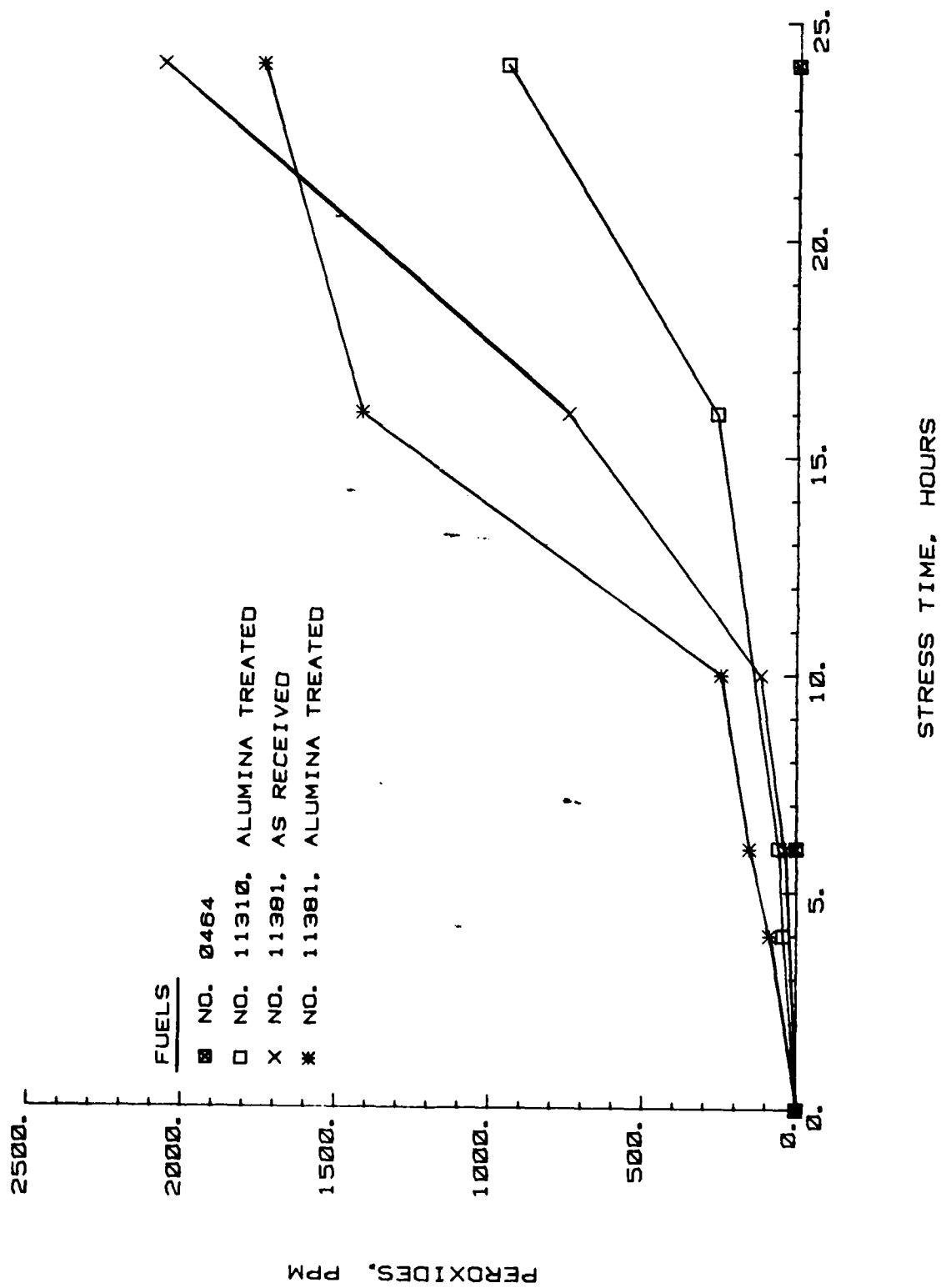


FIGURE 11. OXIDATION AT 100°C WITH 100 PSI OXYGEN (PEROXIDES, PPM)

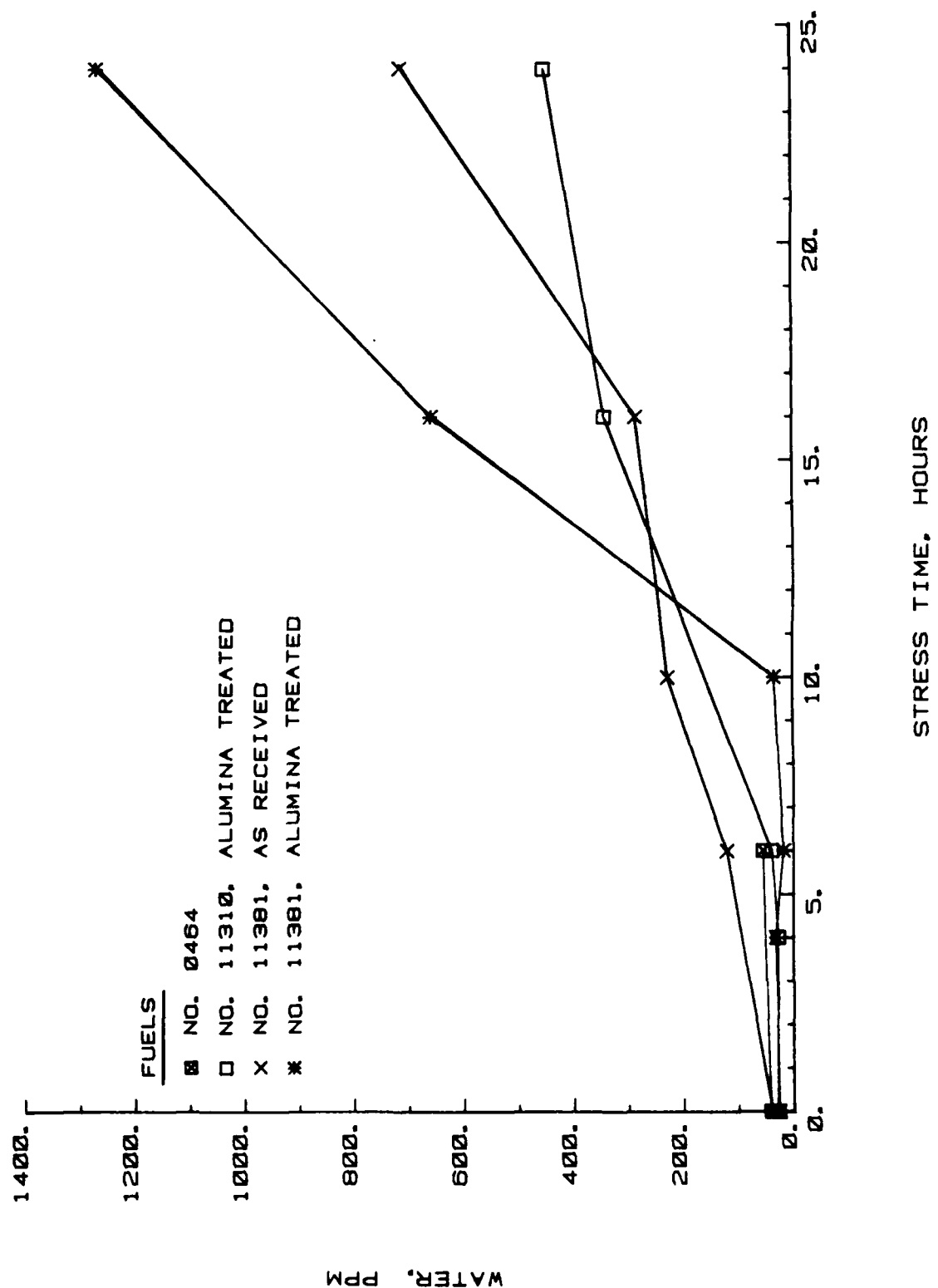


FIGURE 12. OXIDATION AT 100°C WITH 100 PSI OXYGEN (WATER, PPM)

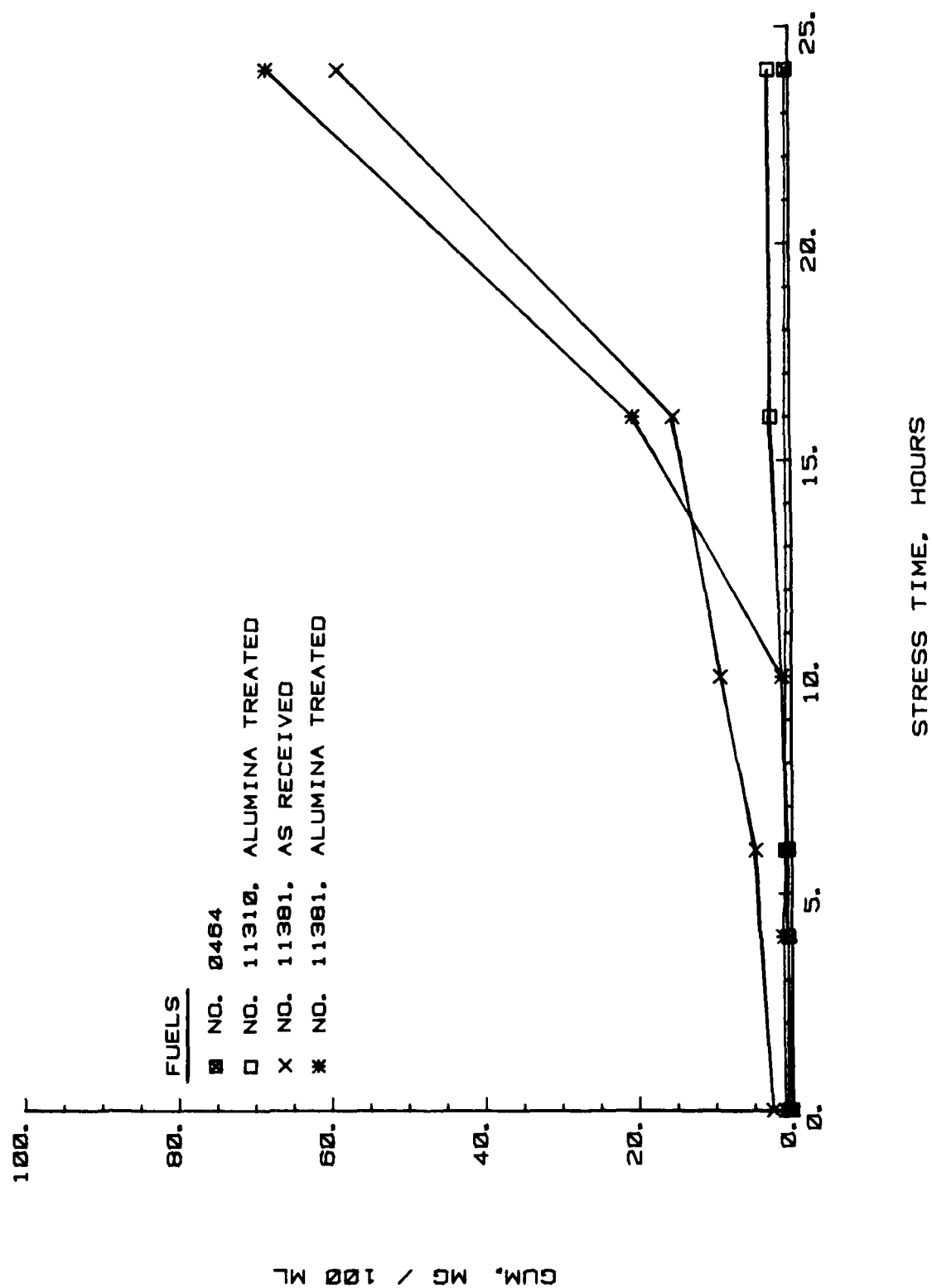


FIGURE 13. OXIDATION AT 100°C WITH 100 PSI OXYGEN (GUM, MG/100 ML)

water and gum that were generated, gave inconclusive results. In no instance, were measurable amounts of acidic components produced.

Oxidation of the same kerosenes at 100°C under the influence of 689 kPa (100 psig) of oxygen (99.99%) produced substantial degradation while producing only small amounts of acidic components. When the results of these experiments were compared with those of the bottle storage, it was apparent that both methods agree on the identity of the most stable fuel (No. 0464) and the least stable fuel (No. 11381, alumina-treated). However, the two intermediate fuels' positions have been interchanged. No explanation for this observation may be offered at this time.

The tentative relative ranking of the four fuels after ultimate indicated stress periods by the three test procedures are summarized in Table 7.

A further observation may also be made from the pressure gauge readings during test, as given under the heading of "Oxygen Depletion" in Table 4 and 5. As discussed previously, the reactor was pressurized to a predetermined extent, e.g., 100 psig, at a measured room temperature. Upon completion of the test, the reactor and its contents were cooled to room temperature, and pressure and temperature readings were taken again. From these data, the total amount of consumed oxygen was calculated. During the test, a continuous recording was made of the reactor pressures. As the reactor was heated from room temperature, e.g., 23° to 100°C, the pressure inside the reactor should have increased from 689 kPa (100 psig) to 896 kPa (130 psig). It was found, however, that a maximum pressure of 800 kPa (116 psig) developed 1 hour after the reactor reached the set temperature of 100°C. The average initial pressure was 758 kPa (110 psig) with a standard deviation of 17.2 kPa (2.5 psig). Similarly, at 60°C the calculated oxygen pressure of 786 kPa (114 psig) was higher than the observed pressures given in Table 4. In both cases, the balance of the oxygen was assumed to be the increased amount of oxygen that dissolved in the fuel due to the increase in temperature.

TABLE 7. RANKING OF FUELS ACCORDING TO VARIOUS TESTS

Property	Fuel Identification Number				
	"Stable Fuel"			"Unstable Fuel"	
<u>Bottle Storage: 43°C/24 weeks</u>					
Oxygen in vapor phase	0464		11381	11310A	11381A
Oxygen in liquid phase	0464		11381	11310A	11381A
Peroxide content	0464		11381	11310A	11381A
Water content	0464	≈	11381	11310A	11381A
Gum content	0464	≈	11310A	11381	11381A
<u>Reactor: 60°C/100 PSIG/48 hours</u>					
Oxygen consumed	0464	≈	11381	11381A	11310A
Peroxide content	0464	≈	11381	11310A	11381A
Water content	11381		11381A	11310	0464
Gum content	11310A		0464	11381A	11381
<u>Reactor: 100°C/100 PSIG/24 hours</u>					
Oxygen consumed	0464		11310A	11381	11381A
Peroxide content	0464		11310A	11381A	11381
Water content	--		11310A	11381	11381A
Gum content	0464	≈	11310A	11381	11381A

V. CONCLUSIONS AND RECOMMENDATIONS

Through the generally accepted 43°C (110°F) bottle storage method of accelerated fuel aging, the relative ratings of four selected fuels' oxidative tendencies were established. In this method, storage for about 12 weeks produced results comparable to a full year's storage under ambient conditions. To develop a practical test method for the prediction of peroxide potential of fuels, experimental conditions were sought that the oxidative tendencies of fuels could be assessed within a reasonable time, i.e., less than 48 hours. Accordingly, a matrix of experiments was designed to allow selection of those reaction conditions that would give results comparable to the 43°C bottle storage experiments. Additionally, the results of these experiments would allow the development of global reaction kinetics to aid the determination of fuel peroxidation potential. From the partially completed experimental matrix (as defined earlier), fuel stressing was completed at 60° and 100°C under an initial oxygen pressure of 689 kPa (100 psig). Experimental results at 60°C gave inconclusive results. Results of the 100°C experiments produced essentially self-consistent results that also agreed with those of the bottle storage for the most stable and least stable fuels. Results of the two intermediate stability fuels, however, were interchanged.

It is recommended that the proposed experimental matrix be completed to allow completion of project objectives. However, as a consequence of inconclusive findings at 60°C and 689 kPa (100 psig), it is recommended that the original matrix be modified to delete the 345 kPa (50 psig) condition at 60°C. This work should then be followed by determination of reliability and accuracy of this possible new practical analytical method.

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3. Proposed ASTM Test Method for Distillate Fuel Storage Stability at 43°C .

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DOVER NJ 07801-5001

CDR, US ARMY TROOP SUPPORT
COMMAND
ATTN: AMSTR-ME 1
AMSTR-S 1
4300 GOODFELLOW BLVD
ST LOUIS MO 63120

CDR
CONSTRUCTION ENG RSCH LAB
ATTN: CERL-EM 1
CERL-ZT 1
CERL-EH 1
P O BOX 4005
CHAMPAIGN IL 61820

TRADOC LIAISON OFFICE
ATTN: ATFE-LO-AV 1
4300 GOODFELLOW BLVD
ST LOUIS MO 63120

CDR
11TH TRANSPORTATION BATTALION
(TERMINAL)
ATTN: AFFG-I-CDR 1
FORT STORY VA 23459

HQ
US ARMY TRAINING & DOCTRINE CMD
ATTN: ATCD-SL-5 (MAJ JONES) 1
FORT MONROE VA 23651-5000

DIRECTOR
US ARMY RSCH & TECH LAB
(AVRADCOM)
PROPULSION LABORATORY
ATTN: SAVDL-PL-D (MR ACURIO) 1
21000 BROOKPARK ROAD
CLEVELAND OH 44135

CDR
US ARMY NATICK RES & DEV LAB
ATTN: STRNA-YE (DR KAPLAN) 1
STRNA-U 1
NATICK MA 01760

CDR
US ARMY TRANSPORTATION SCHOOL
ATTN: ATSP-CD-MS (MR HARNET) 1
FORT EUSTIS VA 23604

PROJ MGR, PATRIOT PROJ OFFICE
ATTN: AMCPM-MD-T-C 1
U.S. ARMY MISSILE COMMAND
REDSTONE ARSENAL AL 35809

CDR
US ARMY QUARTERMASTER SCHOOL
ATTN: ATSM-CD 1
ATSM-TD 1
ATSM-PFS 1
FORT LEE VA 23801

HQ, US ARMY ARMOR CENTER AND
FORT KNOX
ATTN: ATSB-CD 1
FORT KNOX KY 40121

CDR
101ST AIRBORNE DIV (AASLT)
ATTN: AFZB-KE-J 1
AFSB-KE-DMMC 1
FORT CAMPBELL KY 42223

CDR
US ARMY WESTERN COMMAND
ATTN: APLG-TR
FORT SCHAFTER HI 96858

CDR
COMBINED ARMS COMBAT
DEVELOPMENT ACTIVITY
ATTN: ATZL-CAT-E
ATZL-CAT-A
FORT LEAVENWORTH KA 66027-5300

CDR
US ARMY LOGISTICS CTR
ATTN: ATCL-MS (MR A MARSHALL)
ATCL-C
FORT LEE VA 23801-6000

PROJECT MANAGER
PETROLEUM & WATER SYSTEMS
ATTN: AMCPM-PWS (LTC FOSTER)
4300 GOODFELLOW BLVD
ST LOUIS MO 63120

CDR
US ARMY FIELD ARTILLERY SCHOOL
ATTN: ATSF-CD
FORT SILL OK 73503-5600

CDR
US ARMY ENGINEER SCHOOL
ATTN: ATZA-TSM-G
ATZA-CDM
ATZA-CDD
FORT BELVOIR VA 22060-5606

CDR
US ARMY INFANTRY SCHOOL
ATTN: ATSH-CD-MS-M
FORT BENNING GA 31905-5400

CDR
MILITARY TRAFFIC MANAGEMENT
COMMAND
ATTN: MT-SA (MR DOWD)
WASHINGTON DC 20315

DIR
US ARMY MATERIALS & MECHANICS
RESEARCH CENTER
ATTN: AMXMR-M
AMXMR-O
WATERTOWN MA 02172-2796

CDR
US ARMY MISSILE CMD
ATTN: AMSMI-U
AMSMI-RR
AMSMI-S
REDSTONE ARSENAL AL 35898-5242

CDR
US ARMY AVIATION CTR & FT RUCKER
ATTN: ATZQ-DI
FORT RUCKER AL 36362

PROG MGR, TANK SYSTEMS
ATTN: AMCPM-MIEI-SM
AMCPM-M60
WARREN MI 48397

CDR
US ARMY ARMOR & ENGINEER BOARD
ATTN: ATZK-AE-AR
ATZK-AE-LT
FORT KNOX KY 40121

CDR
6TH MATERIEL MANAGEMENT CENTER
19TH SUPPORT BRIGADE
APO SAN FRANCISCO 96212

CHIEF, U.S. ARMY LOGISTICS
ASSISTANCE OFFICE, FORSCOM
ATTN: AMXLA-FO (MR PITTMAN)
FT MCPHERSON GA 30330

CDR
US ARMY SAFETY CENTER
ATTN: PESC-SSD (MR BUCHAN)
FORT RUCKER AL 36362

DEPARTMENT OF THE NAVY

CDR
NAVAL AIR PROPULSION CENTER
ATTN: PE-33 (MR D'ORAZIO)
PE-32 (MR MANGIONE)
P O BOX 7176
TRENTON NJ 06328

CDR
NAVAL SEA SYSTEMS CMD
ATTN: CODE 05M4 (MR R LAYNE)
WASHINGTON DC 20362

CDR
DAVID TAYLOR NAVAL SHIP R&D CTR
ATTN: CODE 2830 (MR BOSMAJIAN) 1
CODE 2759 (MR STRUCKO) 1
CODE 2831 1
ANNAPOLIS MD 21402

CG
FLEET MARINE FORCE ATLANTIC
ATTN: G4 (COL ROMMANTZ) 1
NORFOLK VA 23511

CDR
NAVAL SHIP ENGINEERING CENTER
ATTN: CODE 6764 (MR. BOYLE) 1
PHILADELPHIA PA 19112

JOINT OIL ANALYSIS PROGRAM -
TECHNICAL SUPPORT CTR 1
BLDG 780
NAVAL AIR STATION
PENSACOLA FL 32508

PROJ MGR, M60 TANK DEVELOPMENT
ATTN: USMC-LNO 1
US ARMY TANK-AUTOMOTIVE
COMMAND (TACOM)
WARREN MI 48397

DEPARTMENT OF THE NAVY
HQ, US MARINE CORPS
ATTN: LPP (MAJ WALLER) 1
LMM/3 (MAJ WESTERN) 1
WASHINGTON DC 20380

CDR
NAVAL AIR SYSTEMS CMD
ATTN: CODE 53645 (MR MEARNES) 1
WASHINGTON DC 20361

CDR
NAVAL AIR DEVELOPMENT CTR
ATTN: CODE 60612 1
WARMINSTER PA 18974

CDR
NAVAL RESEARCH LABORATORY
ATTN: CODE 6170 1
CODE 6180 1
CODE 6110 (DR HARVEY) 1
WASHINGTON DC 20375

CDR
NAVAL FACILITIES ENGR CTR
ATTN: CODE 1202B (MR R BURRIS) 1
200 STOWWALL ST
ALEXANDRIA VA 22322

CDR
NAVAL AIR ENGR CENTER
ATTN: CODE 92727 1
LAKEHURST NJ 08733

COMMANDING GENERAL
US MARINE CORPS DEVELOPMENT
& EDUCATION COMMAND
ATTN: DO74 (LTC WOODHEAD) 1
QUANTICO VA 22134

OFFICE OF CHIEF OF NAVAL
RESEARCH
ATTN: ONT-07E (MR ZIEM) 1
ARLINGTON, VA 22217

CHIEF OF NAVAL OPERATIONS
ATTN: OP 413 1
WASHINGTON DC 20350

GG
FLEET MARINE FORCE PACIFIC
ATTN: G4 (COL HARMS) 1
CAMP H.M. SMITH HI 96861

CDR
NAVY PETROLEUM OFC
ATTN: CODE 43 1
CAMERON STATION
ALEXANDRIA VA 22314

DEPARTMENT OF THE AIR FORCE

HQ, USAF
ATTN: LEYSF (COL CUSTER) 1
WASHINGTON DC 20330

HQ AIR FORCE SYSTEMS CMD
ATTN: AFSC/DLF (MAJ VONEDA) 1
ANDREWS AFB MD 20334

CDR
US AIR FORCE WRIGHT AERONAUTICAL
LAB
ATTN: AFWAL/POSF (MR CHURCHILL) 1
AFWAL/POSL (MR JONES) 1
AFWAL/MLSE (MR MORRIS) 1
WRIGHT-PATTERSON AFB OH 45433

CDR
SAN ANTONIO AIR LOGISTICS
CTR
ATTN: SAALC/SFT (MR MAKRIS) 1
SAALC/MMPRR 1
KELLY AIR FORCE BASE TX 78241

CDR
WARNER ROBINS AIR LOGISTIC
CTR
ATTN: WRALC/MMTV (MR GRAHAM) 1
ROBINS AFB GA 31098

CDR
USAF 3902 TRANSPORTATION
SQUADRON
ATTN: LGTVP (MR VAUGHN) 1
OFFUTT AIR FORCE BASE NE 68113

CDR
HQ 3RD USAF
ATTN: LGSF (MR PINZOLA) 1
APO NEW YORK 09127

CDR
DET 29
ATTN: SA-ALC/SFM 1
CAMERON STATION
ALEXANDRIA VA 22314

OTHER GOVERNMENT AGENCIES

NATIONAL AERONAUTICS AND
SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
MAIL STOP 5420
(ATTN: MR. GROBMAN) 1
CLEVELAND OH 44135

NATIONAL AERONAUTICS AND
SPACE ADMINISTRATION
VEHICLE SYSTEMS AND ALTERNATE
FUELS PROJECT OFFICE
ATTN: MR CLARK 1
LEWIS RESEARCH CENTER
CLEVELAND OH 44135

DEPARTMENT OF TRANSPORTATION
FEDERAL AVIATION ADMINISTRATION
ATTN: AWS-110 1
800 INDEPENDENCE AVE, SW
WASHINGTON DC 20590

US DEPARTMENT OF ENERGY
CE-1312
ATTN: MR ECKLUND 1
FORRESTAL BLDG.
1000 INDEPENDENCE AVE, SW
WASHINGTON DC 20585

ENVIRONMENTAL PROTECTION
AGENCY
AIR POLLUTION CONTROL 1
2565 PLYMOUTH ROAD
ANN ARBOR MI 48105

AGENCY FOR INTERNATIONAL
DEVELOPMENT
ATTN: MR D HOOKER 1
M/SER/EOMS/OPM, ROOM 2155A11
WASHINGTON DC 20523

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